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Indian Standard
SPECIFICATION FOR
RESORCINOL

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INDIAN STANDARDS INSTITUTION MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

Indian Standard SPECIFICATION FOR RESORCINOL

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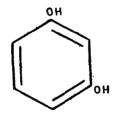
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Indian Standard SPECIFICATION FOR RESORCINOL

0. FOREWORD

- 0.1 This Indian Standard was adopted by the Indian Standards Institution on 8 December 1983, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.
- 0.2 Resorcinol or resorcine, $C_6H_4(OH)_2$, is chemically described as 3-hydroxy phenol which is white crystals becoming pink on exposure to light. It is used in the manufacture of styphnic acid tanning agents, synthetic resin adhesives, pharmaceuticals, dyes, celluloid (camphor substitute) and rubber tackifiers. It is represented by the following structural formula.



RESORCINOI.
(MOLECULAR MASS 110.11)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for resorcinol.

^{*}Rules for rounding off numerical values (revised).

2. REQUIREMENTS

- **2.1 Description** The material shall consist essentially of m-dihydroxy benzene in the form of clean white flakes or crystalline powder/lumps. When melted, the melt shall have a colour not darker than pale straw and shall be free from grit and suspended matter.
- 2.2 The material shall also comply with the requirements given in Table 1 when tested according to the test methods prescribed in Appendix A. Reference to the relevant clauses of the appendix is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR RESORCINOL						
S _L No.	Characteristic	REQUIREMENT	METHOD OF TEST (REF TO CL NO. IN APPENDIX A)			
(1)	(2)	(3)	(4)			
i)	Crystallizing point, °C, Min	109.0	A-2			
ii)	Moisture, percent by mass, Max	0.5	A-3			
iii)	Purity, percent by mass, Min	98·5	A-4			
i v)	Sulphated ash, percent by mass, Max	0.1	A-5			
v)	Free phenol	To pass the test	A-6			
vi)	Matter insoluble in water, percent by mass, Max	0.03	A-7			

3. PACKING AND MARKING

- 3.1 Packing The material shall be suitably packed to protect against sunlight and moisture. The quantity per package shall be 25 kg or as agreed to between the supplier and the purchaser.
- 3.2 Marking Each container shall be securely closed and shall bear legibly and indelibly the following information:
 - a) Name of the material;
 - b) Name of the manufacturer and his trade-mark, if any;
 - c) Lot or batch number in code or otherwise; and
 - d) Gross, net and tare mass.

3.2.1 The containers may also be marked with the ISI Certification Mark.

Note—The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix B.

APPENDIX A

(Clause 2.2)

METHODS OF TEST FOR RESORCINOL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS: 1070-1977*) shall be employed in the tests.

Note—'Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF CRYSTALLIZING POINT

A-2.1 Procedure—Carry out the determination of crystallizing point according to the method as prescribed in IS: 5813-1970†.

A-3. DETERMINATION OF MOISTURE

A-3.1 Carry out the determination of moisture according to the method as prescribed in IS: 2362-1973‡.

A-4. DETERMINATION OF PURITY

A-4.1 Reagents

^{*}Specification for water for general laboratory use (second rivision).

[†]Method for determination of crystallizing point.

[‡]Determination of water by Karl Fischer method (first rivision).

- A-4.1.1 Concentrated Sulphuric Acid See IS: 266-1977*.
- A-4.1.2 Standard Bromate Bromide Solution 0.1 N. Dissolve 2.784 g of potassium bromate and 12 g of potassium bromide in 1000 ml of water.
 - A-4.1.3 Concentrated Hydrochloric Acid See IS: 265-1976.
 - A-4.1.4 Potassium Iodide Solution 10 percent.
 - A-4.1.5 Standard Sodium Thiosulphate Solution 0.1 N.
 - A-4.1.6 Starch Indicator Solution 1 percent.
- A-4.2 Procedure—Dissolve 0.7 g of the material in water and dilute to 500 ml. Pipette 50 ml into a glass-stoppered flask, dilute with 25 ml of water and add 50 ml of 0.1 N bromate—bromide solution and 10 ml of 5 N hydrochloric acid. Stopper this flask, shake and allow to stand for 5 minutes in the dark. Add 20 ml of 10 percent potassium iodide solution and titrate at once with 0.1 N sodium thiosulphate, using starch solution as indicator.

A-4.3 Calculation

Resorcinol content, percent by mass = $\frac{0.1835 \times V}{M}$

where

V=volume in ml of standard sodium thiosulphate used in the titration, and

M=mass in g of the material taken for the test.

A-5. DETERMINATION OF SULPHATED ASH

A-5.1 Apparatus

- A-5.1.1 Muffle Furnace capable of maintaining temperature within 800±25°C.
 - A-5.1.2 Crucible of silica or platinum, having a capacity of 100 ml.
 - A-5.1.3 Desiccator containing suitable desiccant.

A-5.2 Reagents

A-5.2.1 Concentrate Sulphuric Acid

^{*}Specification for sulphuric acid (second revision).

[†]Specification for hydrochloric acid (second revision).

A-5.3 Procedure — Heat the crucible in the muffle furnance at 800 $\pm 25^{\circ}$ C for 10 to 15 minutes, allow to cool sufficiently to enable it to be transferred to a desiccator. When cool, weigh it to the nearest 0.1 mg. Weigh 5 g of the dried material, accurately to the nearest 0.1 mg into the crucible and moisten with 2 ml of concentrated sulphuric acid. Heat the crucible and its contents gently over a bunsen flame. Allow the material to fume and evaporate to a residue in a well-ventilated fume cupboard. Then ignite the residue in the muffle furnace at $500\pm25^{\circ}$ C until it is free from visible specks of carbon. Cool the crucible in the desiccator and weigh to the nearest 0.1 mg. Repeat the heating for 15 minutes followed by cooling and weighing until two successive weighings do not differ by more than 0.5 mg.

A-5.4 Calculation

Sulphated ash, percent by mass = $\frac{100 M_1}{M_2}$

where

 M_1 = mass in g of the residue, and

 M_2 = mass in g of the material taken for the test.

A-6. TEST FOR FREE PHENOL

A-6.1 Procedure — Make 5 percent solution of the material in a beaker and warm the contents gently. The material shall be taken to have passed the test for free phenol if no odour of phenol is emitted.

A-7. MATTER INSOLUBLE IN WATER

A-7.1 Apparatus

A-7.1.1 Gooch Crucible

A-7.1.2 Oven

A-7.2 Procedure — Dissolve about 5 g of the material in about 500 ml of distilled water in a beaker and stir thoroughly. Heat to boiling if necessary. Cover the beaker with a watch-glass and allow it to stand for 4 hours at room temperature with occasional stirring. Filter the residue in a weighed gooch crucible, applying suction and wash the residue with distilled water. Dry the crucible in an oven maintained at $100\pm2^{\circ}$ C till constant mass is obtained. Weigh the crucible with residue and find out the total matter insoluble in water.

A-7.3 Calculation

Matter insoluble in water, percent by mass $=\frac{M_2-M_1}{M}\times 100$

where

M =mass in g of the sample taken,

 $M_1 = \text{mass in } g \text{ of the crucible, and}$

 M_2 = mass in g of the crucible with residue.

APPENDIX B

(Clause 4.1)

SAMPLING OF RESORCINOL

B-1. SCALE OF SAMPLING

- **B-1.1** Lot All the containers in a single consignment of the material drawn for the same batch of manufacture shall constitute a lot.
- **B-1.2** For ascertaining the conformity of the material in the lot to the requirements of the specification, tests shall be carried out for each lot separately.
- **B-1.3** The number of containers to be selected for this purpose shall depend upon the size of the lot and shall be according to Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE		Z E	Number of Cont. Ters TO BE SELECT
	(1)		(2)
Up	,,	25	3
26	,,	50	4
51	,,	100	5
101	,,	150	6
151	,,	300	7
301	301 and above		8

B-1.3.1 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedures given in IS: 4905-1968* may be followed.

B-2. TEST SAMPLES AND REFEREE SAMPLES

B-2.1 From each of the containers selected according to **B-1.3**, draw with an appropriate sampling instrument small portions of material from different parts of the container. The total quantity so drawn

^{*}Methods for random sampling.

from each of the containers shall be approximately equal to thrice the quantity required for making triplicate determination for all the requirements given in the specification.

- B-2.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.
- B-2.3 From the samples (see B-2.2) representing different containers selected in B-1.3, a small but approximately equal quantity of material shall be taken and thoroughly mixed to form a composite sample of the quantity sufficient to carry out triplicate determinations for the characteristics to be tested on the composite sample (see B-3.2). The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and third for the referee.
- **B-2.4** The remaining portion of the material in the sample (see B-2.2) from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing different containers selected shall be for the purchaser, another for the supplier and the third for the referee.
- B-2.5 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed airtight with stoppers and labelled with full identification particulars such as date of sampling, batch number and other important particulars of the consignment.
- B-2.6 The acteree sample consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of a dispute between the two.

B-3. NUMBER OF TESTS

- B-3.1 Tests for description, crystallizing point and purity shall be conducted on each of the individual samples.
- B-3.2 Tests for determination of remaining requirements given in 2.2 shall be conducted on the composite sample.

B-4. CRITERIA FOR CONFORMITY

- **B-4.1** The lot shall be declared as conforming to the requirements of this specification if **B-4.1.1** and **B-4.1.2** are satisfied.
- B-4.1.1 Each of the test results on the individual samples satisfies the corresponding specification requirement given in 2.2 and Table 1.
- **B-4.1.2** All the test results on the composite sample satisfy the relevant specification requirements given in Table 1.

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